Supercritical Water Mixture (SCWM) Experiment

ISS Flight Experiment using the
High Temperature Insert – Re-flight (HTI-R)
in the DECLIC Facility

June 21, 2012

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Supercritical Water Mixture (SCWM)Experiment

- **The *Supercritical Water Mixture* (SCWM) experiment is a follow-on experiment to the pure *Supercritical Water* HTI experiment conducted on the ISS in 2010**
- **SCWM will use a modified Sample Cell Unit (SCU) filled with a salt-water solution inserted into the refurbished High Temperature Insert (HTI-R) and returned to ISS, ~ March 2013.**

The sample-cell unit (SCU) to be filled with a salt/water mixture

The HTI to be returned from ISS and which houses the SCU

SCWM experiment time-line:

➢ HTI returned to ground ULF-6	Mar 2011
➢ HTI-R refurbishment completed	Sep 2012
➢ HTI-R transfer to KSC	Dec 2012
➢ HTI-R return to ISS	Mar 2013
➢ SCWM ISS testing commences	Jul 2013

Outline:

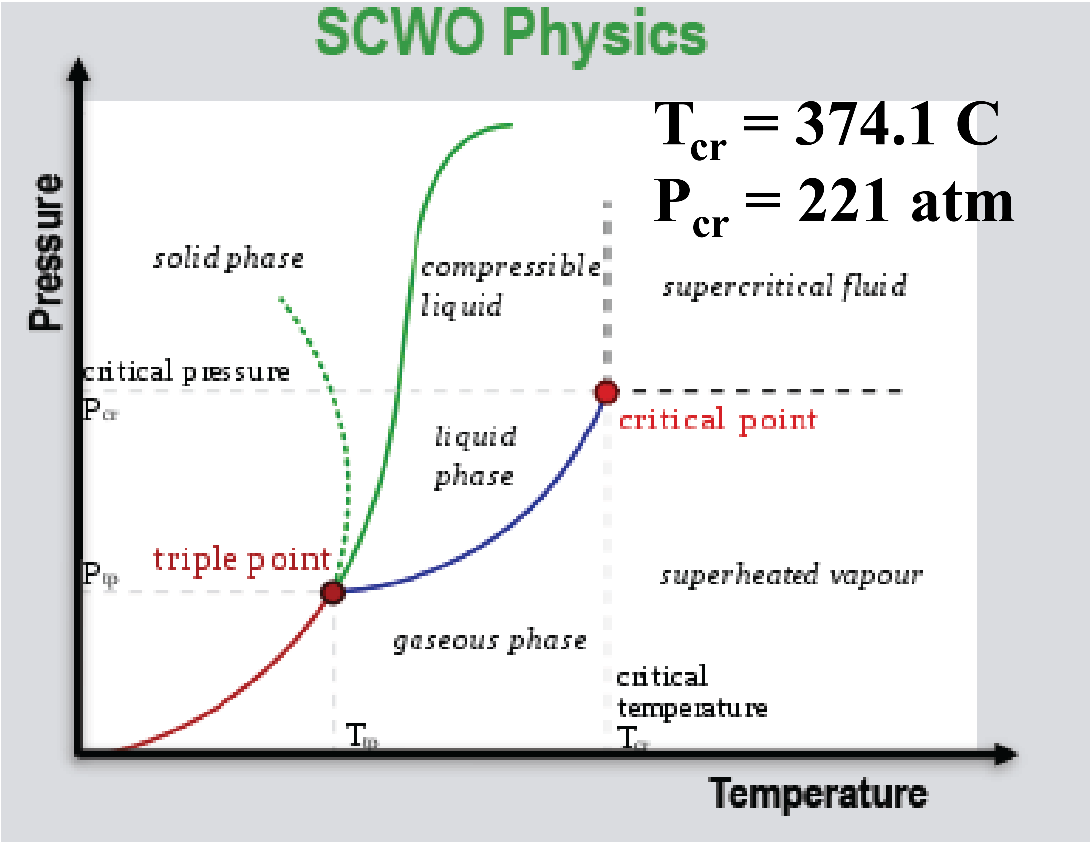
- ***Background***
- ***Motivation***
- ***SCWM Experiment***
  - ***Objectives***
  - ***Salt Selection Criteria***
  - ***Proposed Test Sequences***
- ***Previous ISS Results***
- ***Modeling***
- ***Ground Based Testing***
- ***Acknowledgments***

Background

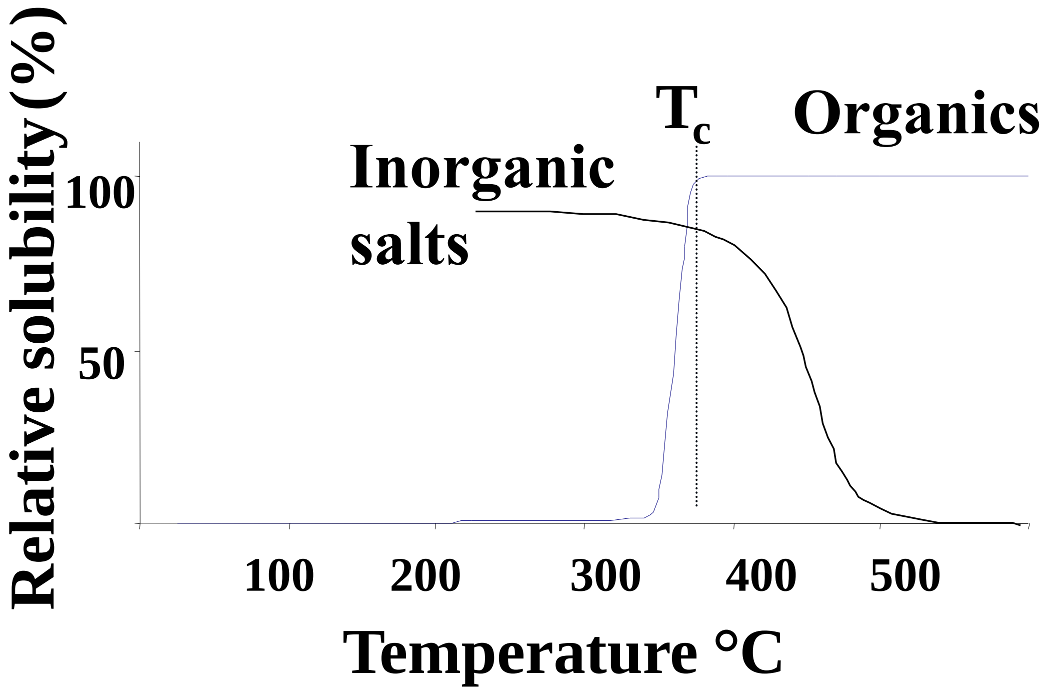
P – T Diagram (pure water)

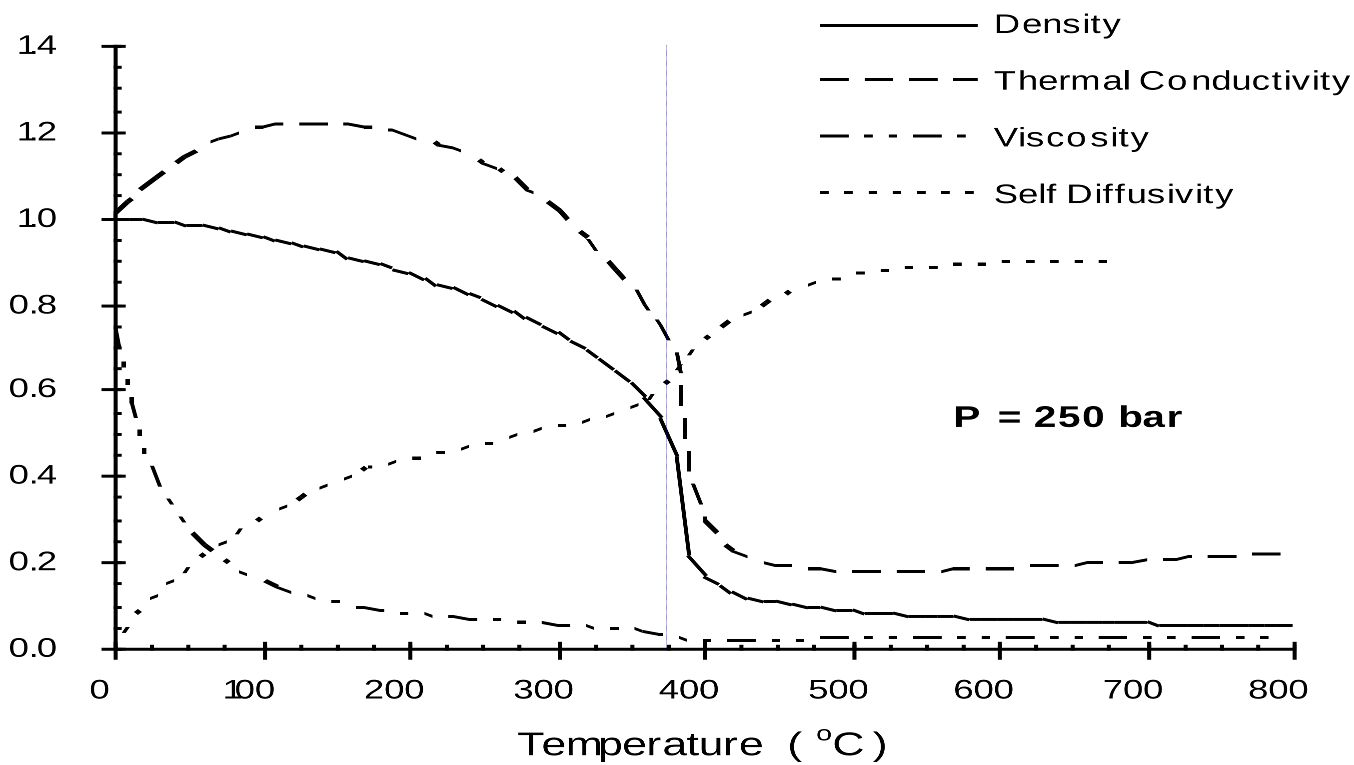
In a supercritical fluid molecular cohesion is liquid- like and molecular diffusion is gas-like.

Supercritical water behaves as a dense, non-polar gas



- **Gases and organic substances are completely soluble in supercritical water**
- **Inorganic salts are insoluble in supercritical water and precipitate out of solution**
- **Oxidizer and hydrocarbon (organic) fuels are brought into intimate single phase contact during reactions**





Between 350 °C and 400 °C (at a pressure of 250 bar)

- **density decreases by 70%**
- **thermal conductivity decreases by 65%**
- **viscosity decreases by 80%**
- **mass diffusivity increases by a factor of 4**

Motivation

NASA's Advanced Exploration

Overarching Goal: ***Develop technologies that promote self sufficiency to enable human exploration beyond low-earth orbit***

Space Studies Board (2000)

Solid waste management: "Both systems (incineration and supercritical water oxidation) show promise for future spacecraft design,"

- **water reclamation from liquid waste streams**
- **CO_2 recovery for O_2 generation and photosynthesis requirements**
- **N_2 recovery from bio-waste streams (plant nutrition recovery for recycling to growth chambers)**
- **transform to beneficial products such as activated carbon, food production substrate, and fuel (from partial oxidation processes)**

2011 NRC Decadal Survey - Super Critical Water Oxidation (SCWO) is strategically aligned with many *"high priority research recommendations"*

Prioritization criteria

(against which high priority research recommendations were assessed)

High priority research recommendations

(w/ SCWO relevancy)

Program Element	Positive Impact on Exploration Efforts, Improved Access to Data or to Samples, Risk reduction	Potential to Enhance Mission Options or to Reduce Mission Costs	Positive Impact on Exploration Efforts, Improved Access to Data or to Samples	Relative Impact within Research Field	Needs Unique to NASA Exploration Programs	Research Programs that could be Dual Use	Research Value of Using Reduced-Gravity Environment	Ability to Translate Results to Terrestrial Needs
Fundamental Physics-Thrust IV: Condensed Matter and Critical Phenomena (FP4)	Low	Low	Low	Medium	Low	Medium	**High**	Medium
Fundamental Fluid Physics: Multi-phase Flows (AP1)	**High**	**High**	**High**	Medium	**High**	Low	**High**	**High**
Fundamental Fluid Physics: Critical Point Phenomena (AP5)	Low	Low	Medium	**High**	Low	Medium	**High**	Medium
Fundamental Combustion Research: Other combustion regimes (AP7)	Medium	Low	Low	**High**	Medium	**High**	**High**	**High**
Translation to Space Exploration Systems: Closed Loop Life Support Systems (TSES6)	Medium	Medium	Medium	Low	**High**	Low	Medium	Low

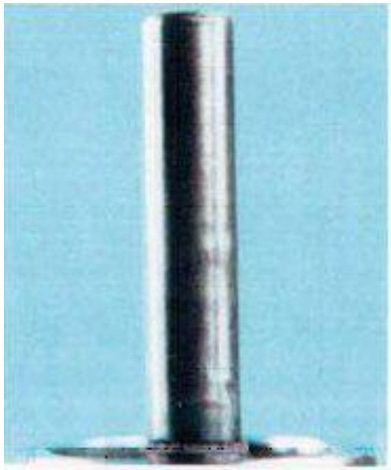
Terrestrial Applications of SCWO

Hirth, Th., et. al., 3rd international symposium on High Pressure Chemical Engineering, Zurich(Switzerland), 1996, p.163

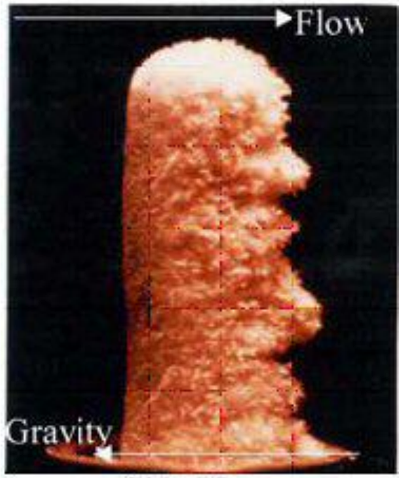
SCWO Unit for Bluegrass Army Depot Richmond, KY

SCWO reactor for municipal sludge: City of Orlando, FL (35 metric tons/day)

- **A key technological hurdle is the control of corrosion and fouling caused by deposition of salt precipitates**
- **This detrimentally impacts operational efficiencies(e.g., heat transfer) and lifetime**
- **Figure[1] provides a dramatic illustration of the rapid deposition on a heated surface inserted into a flowing water/salt solution and maintained at a temperature just above that of the solubility limit in the bulk fluid ($NaSO_4$ at 4 wt % , T_B = 356C, P = 250 atm)**



Before Run

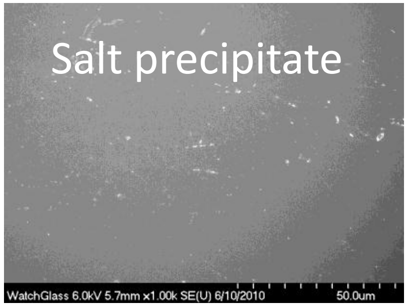


Side View

Hodes, M., Marrone, P., Hong, G.T., Smith, K. A., Tester, J. W., *Salt precipitation and scale control in supercritical water oxidation—Part A: fundamentals and research;* Journal of Supercritical Fluids; 29 (2004) 265–288

- **The SCWM experiment fits naturally in the scheme of investigating supercritical water phenomena … particularly in terms of advancing Supercritical Water Oxidation (SCWO) technology**

Super Critical Water → **Super Critical Water-Salt** → **Super Critical Water Oxidation**



One -> two -> three phase reacting system

ISS Rationale

- **Sharp changes in density and resulting buoyant flow on earth impact ability to study unit processes of salt precipitation and transport**

- **Ground-based reduced-gravity facilities (drop towers, low-g aircraft flights) do not provide sufficient low-g time**

SCWM Experiment Objectives

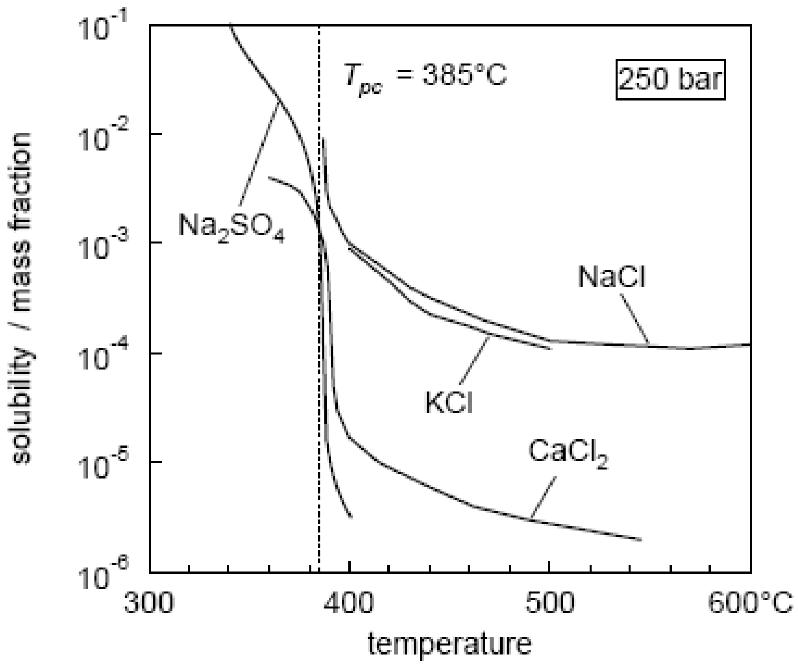
Science Objectives:

The SCWM experiment is designed to study precipitation and transport phenomena of a solute as it precipitates from solution near the critical point of the salt/water mixture.

- **Observe/quantify the critical point of a liquid-gas phase transition for a salt/water mixture**
- **Observe/quantify the onset of precipitation in a supercritical homogeneous phase as a function of temperature**
- **Observe/quantify the transport processes of the precipitate in the presence of temperature and/or salinity gradients**

SCWM Experiment
Salt Selection Criteria

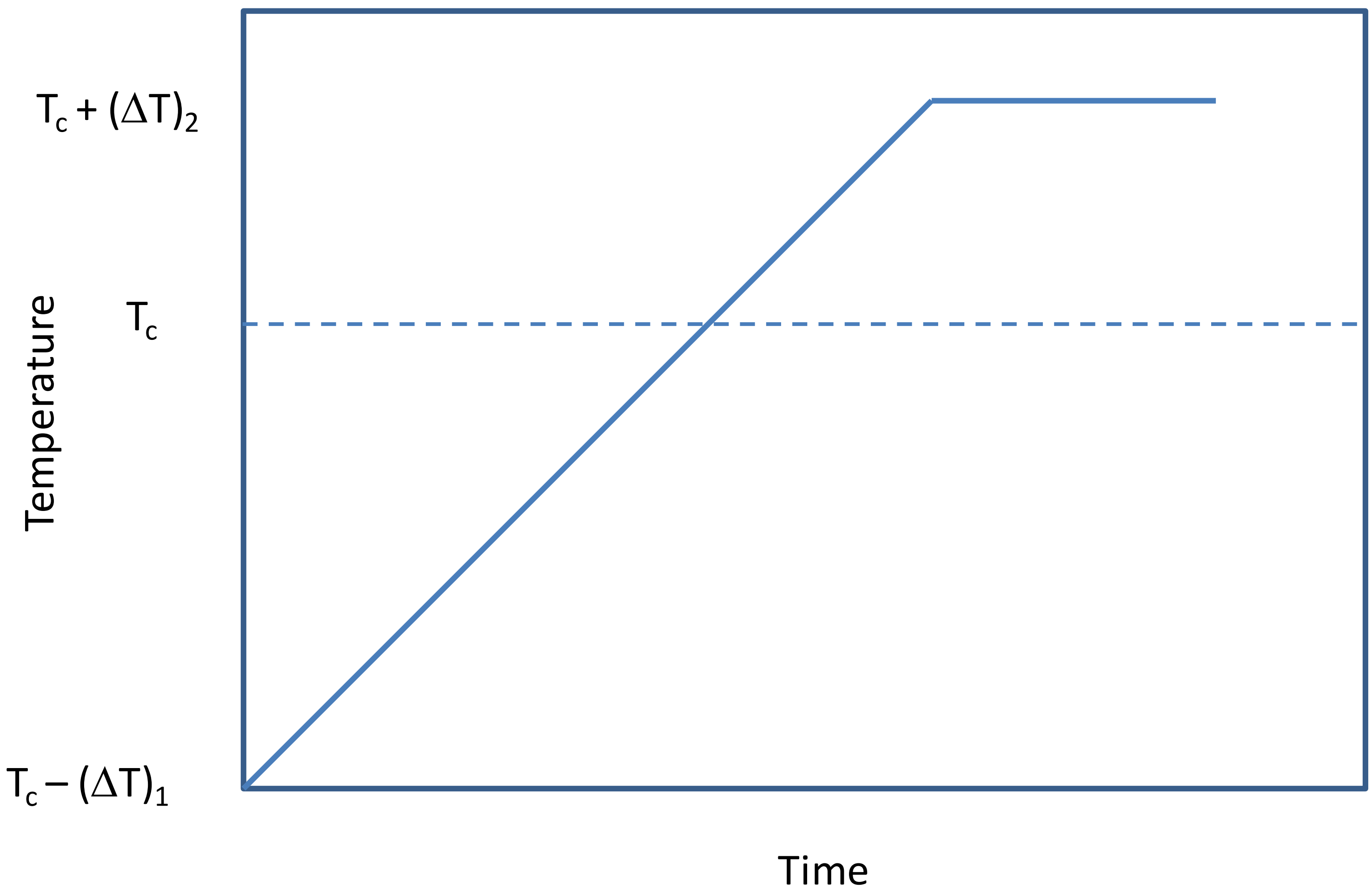
- **Substantial decrease in solubility near critical conditions and below the DECLIC temperature limit (~ 405°C);**
- **Compatible with HTI-R SCU material (Inconel 718)**
- **Available solubility data in water near critical conditions**



SCWM Experiment
Proposed Test Sequences

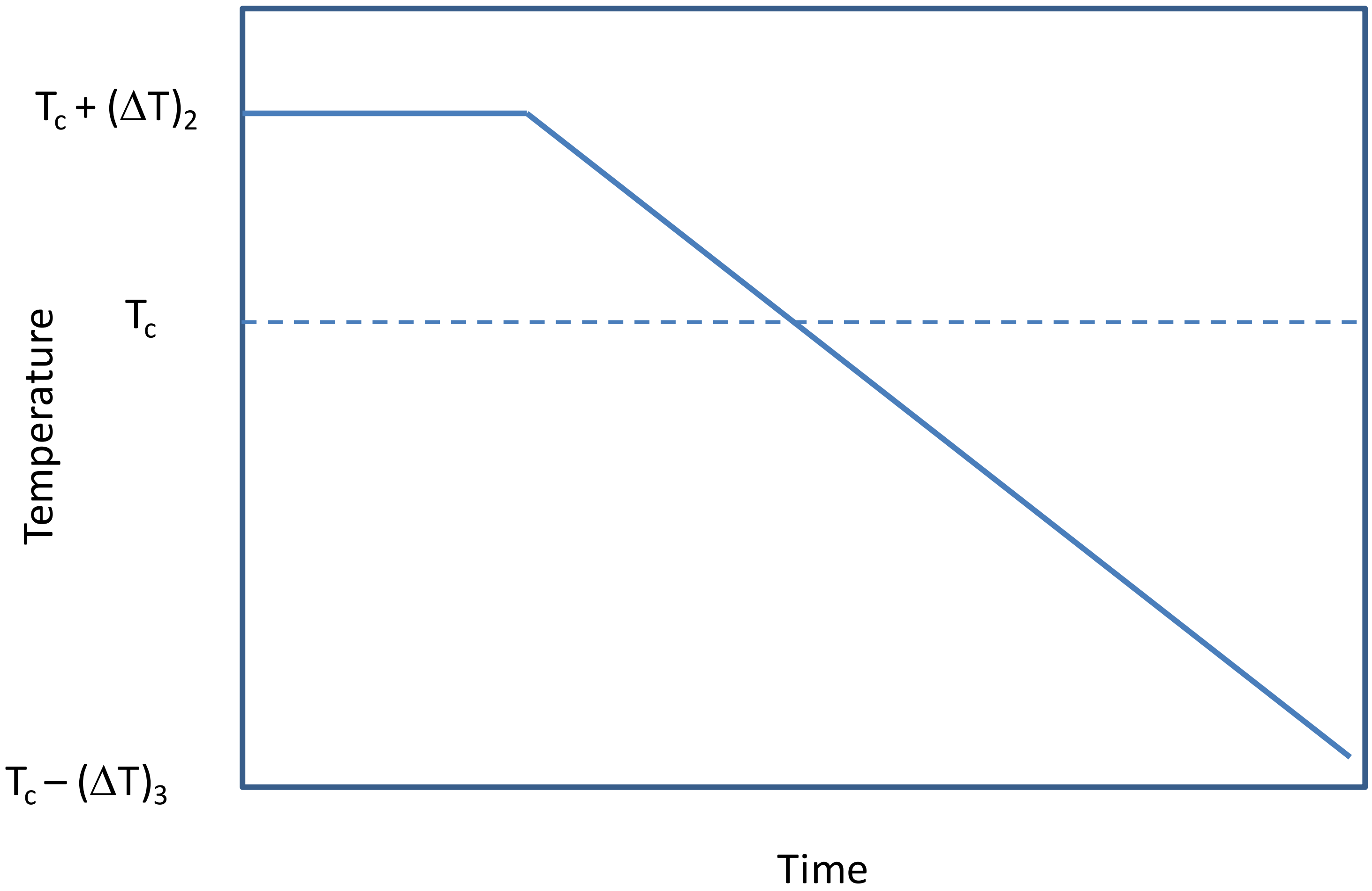
Test Sequence 1

Salt Precipitation During Temperature Increase



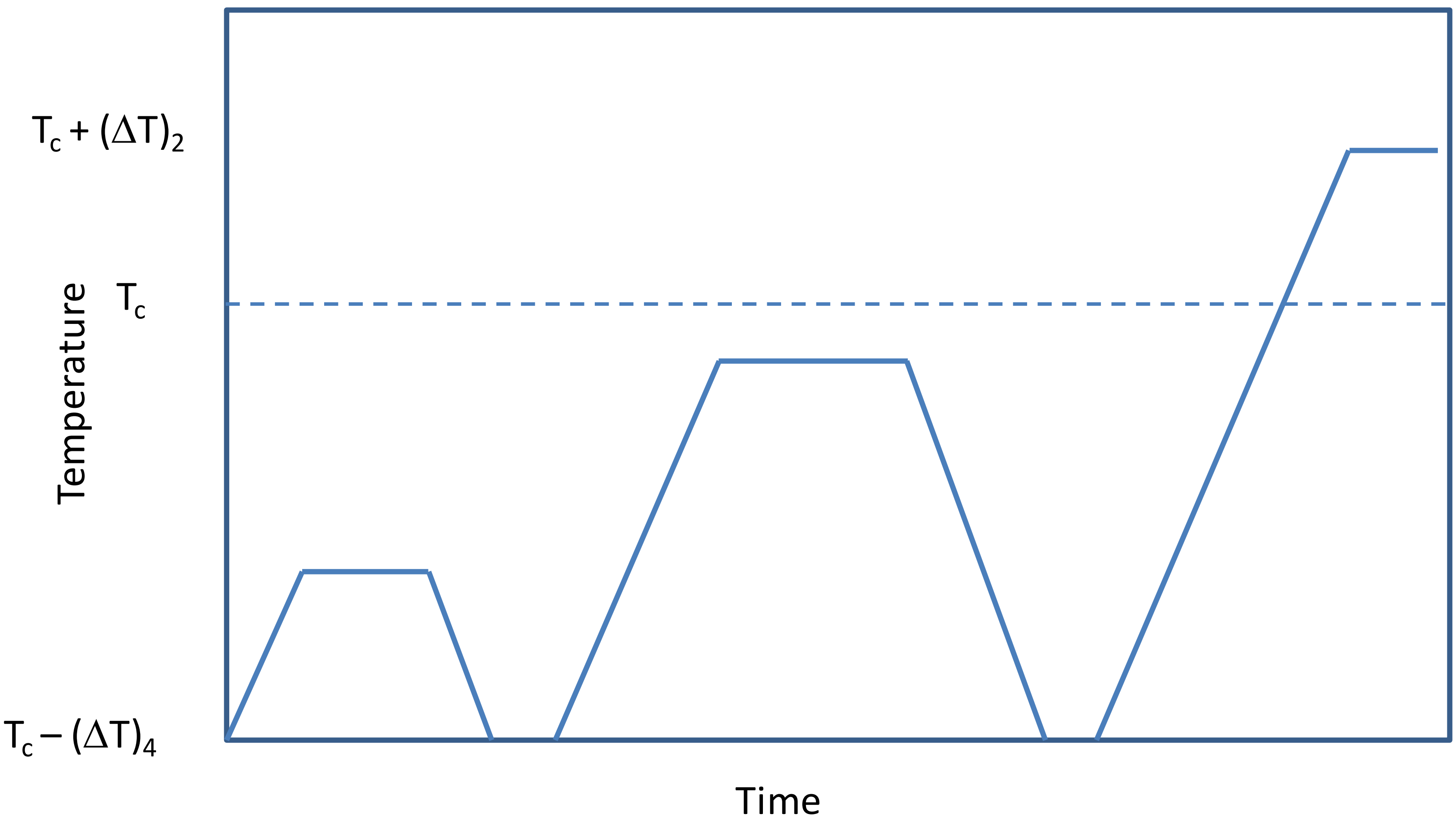
Test Sequence 2

Salt Solvation During Temperature Decrease



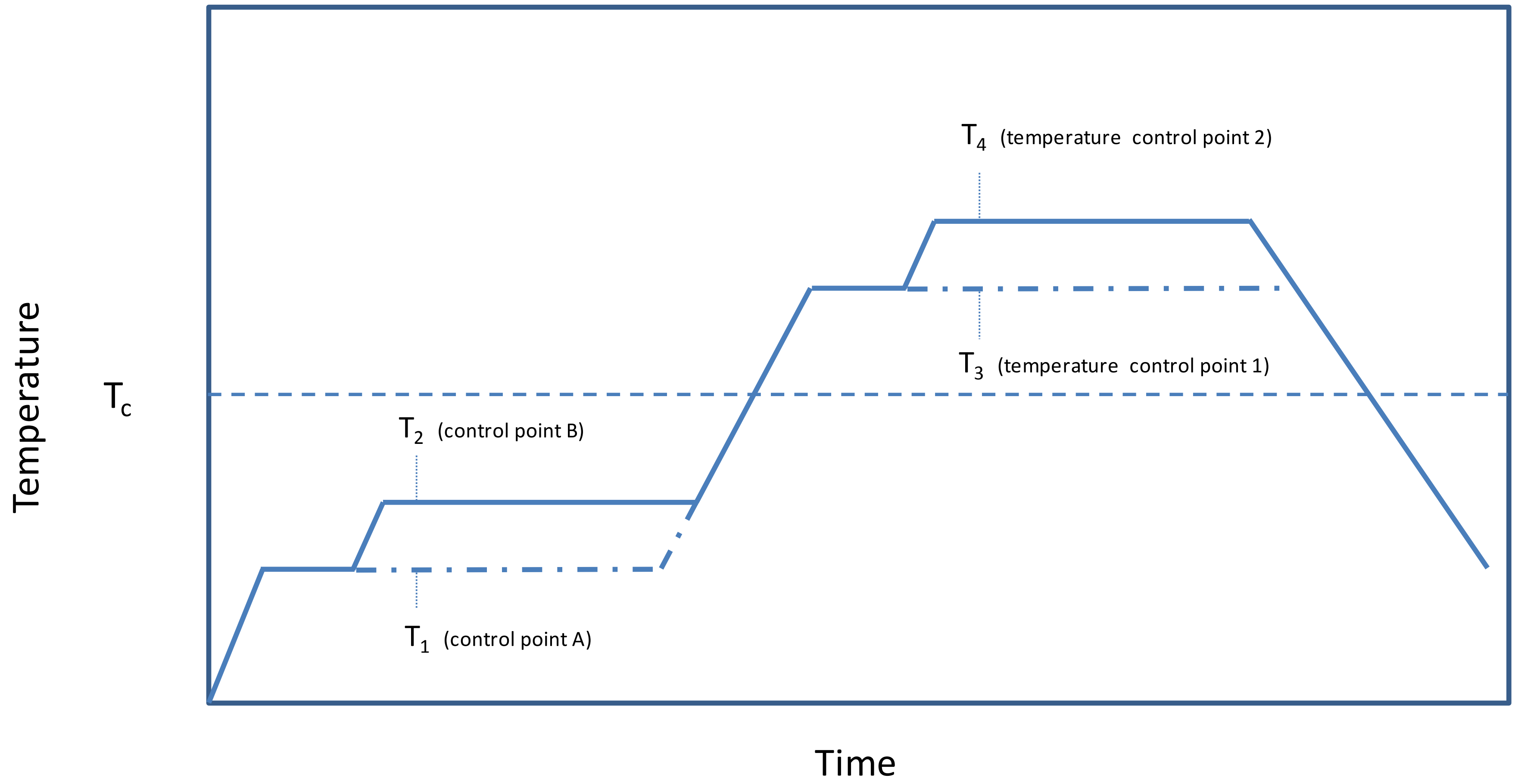
Test Sequence 3

Salt Agglomeration



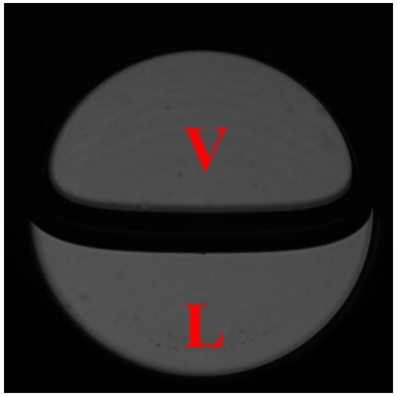
Test Sequence 4

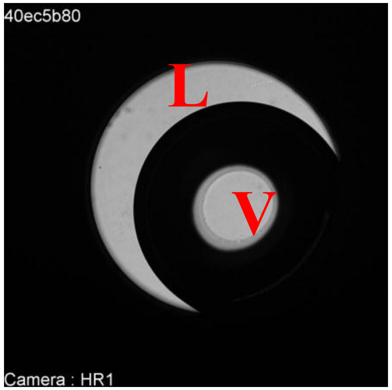
Salt Transport in Near (Sub)-Critical and Supercritical Water



Previous ISS Results
Supercritical Pure Water Studies

Phase Distribution Below the Critical Point





1-g

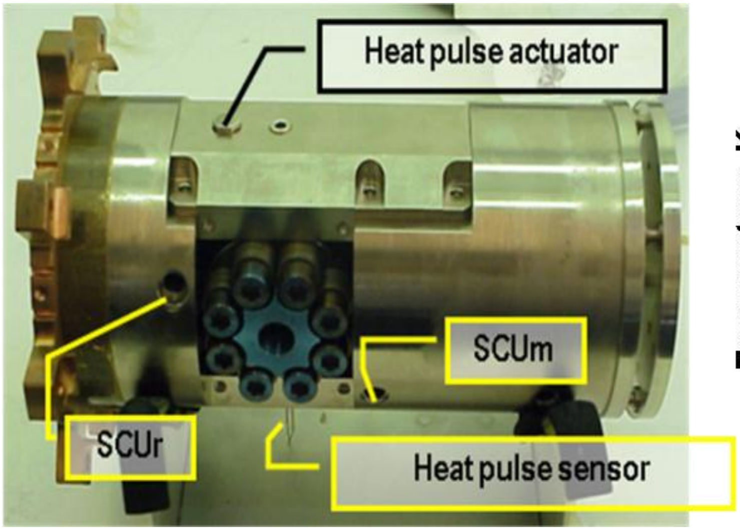
ISS

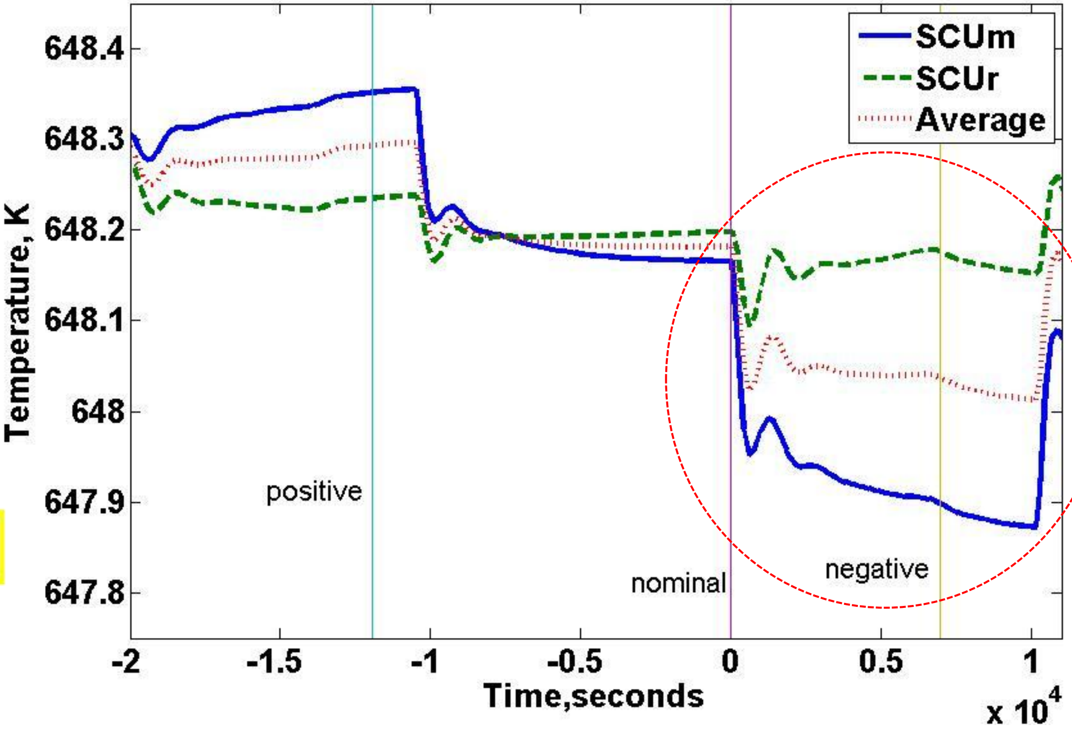
T= 624 K

Dark region between liquid and vapor regions is the meniscus

Analysis

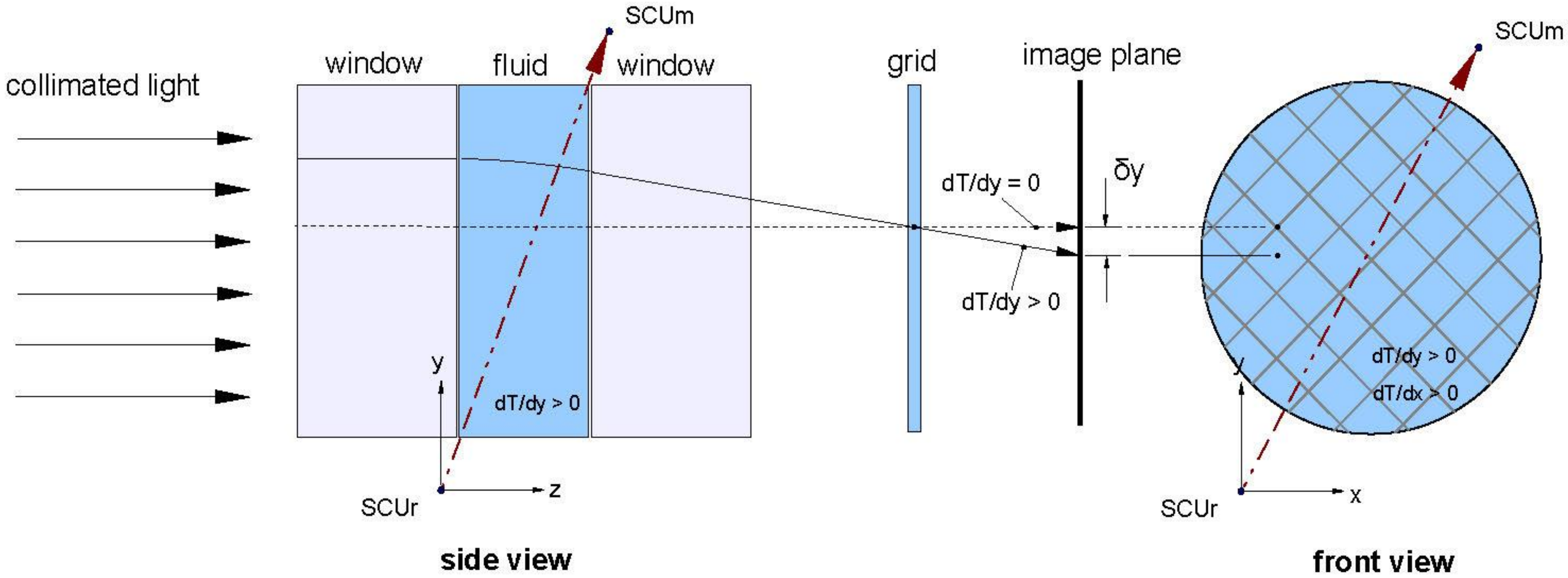
Imposed gradient at $\Delta T \approx$ - 300 mK (with $T \approx T_c + 1$ K)





Analysis (cont)

Shadow-graphic Configuration

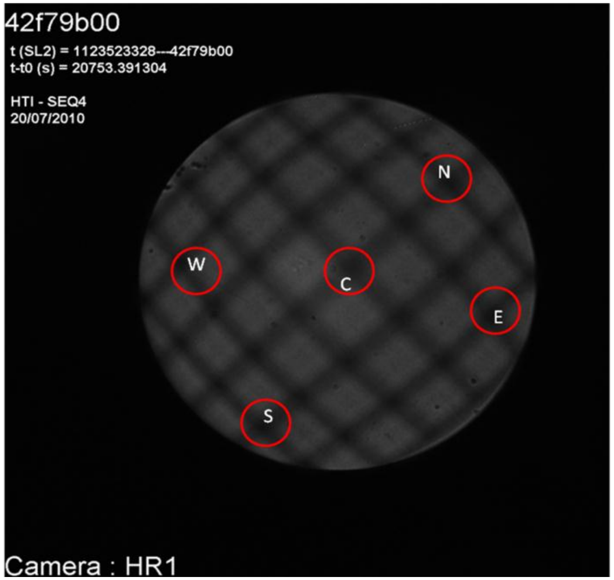


$$\delta = \frac{\psi}{n}\frac{dn}{dy}$$

$$n = 1 + K\rho$$

n = refractive index
ψ , K are constants

Optical Grid Reference Image



T = 648.2 K (~ 1 K above critical point)
Note that there is no vapor/liquid phase difference
Points N, C, W, E, and S are chosen as reference points for tracking the grid displacement under imposed temperature gradients.

Shift Tracking

The shift δ, is expressed as the sum of a spatially uniform component and a local component

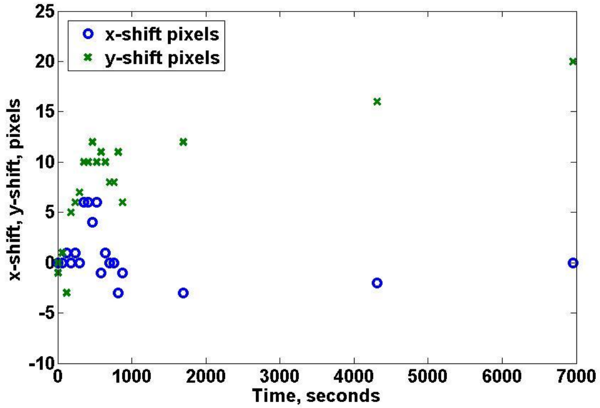
$$\delta = <\delta> + \delta'$$

with

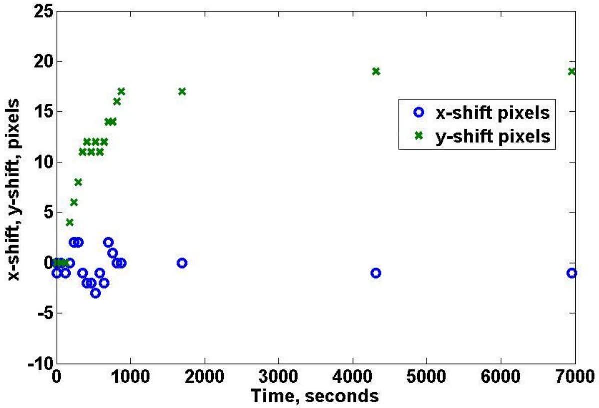
$$<\delta> = \delta(C)$$

The shifts in the reference points at different times during the temperature gradient timeline are tracked using ImageJ. (*Abramoff et al., Biophotonics International, 2004*)

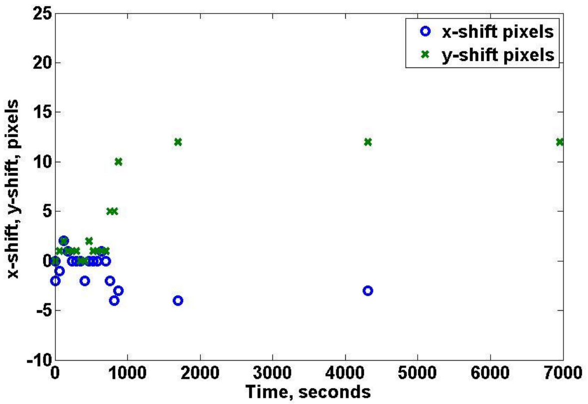
Shift of Reference Points with Time Negative Temperature Gradient



(a) Point N



(b) Point S



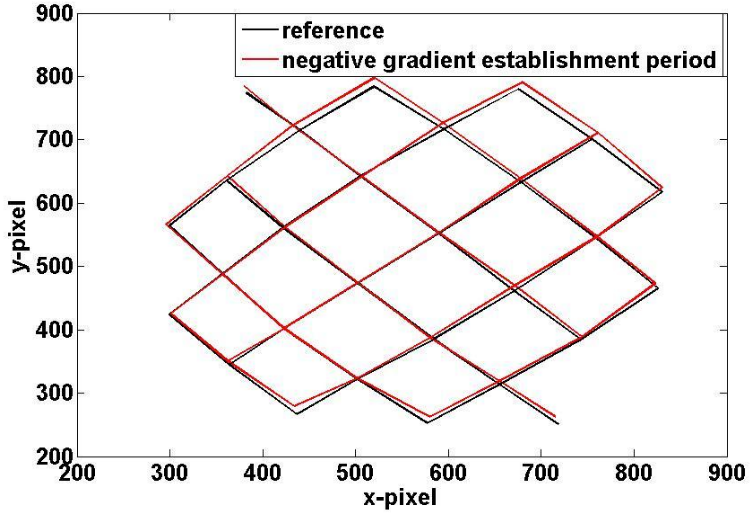
(c) Point C

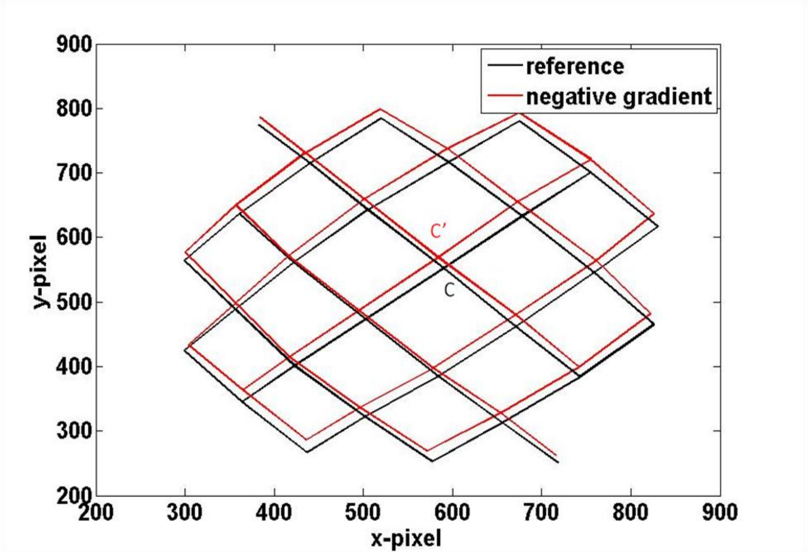
The shift in the y-direction is much greater than the shift in the x-direction.

Shift near the center is delayed compared to near-boundary points.

This is a consequence of reduced thermal diffusivity near the critical point.

Grid Shift for Negative Temperature Gradient

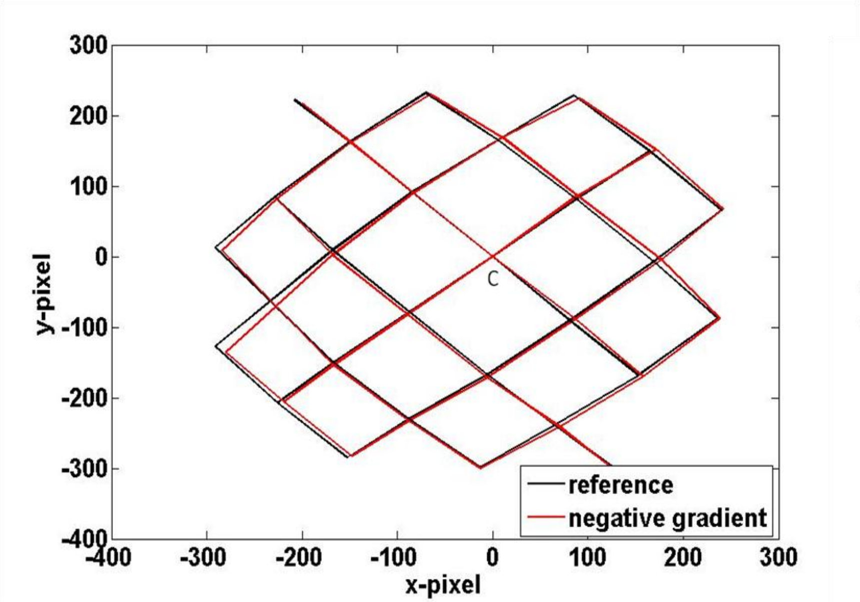


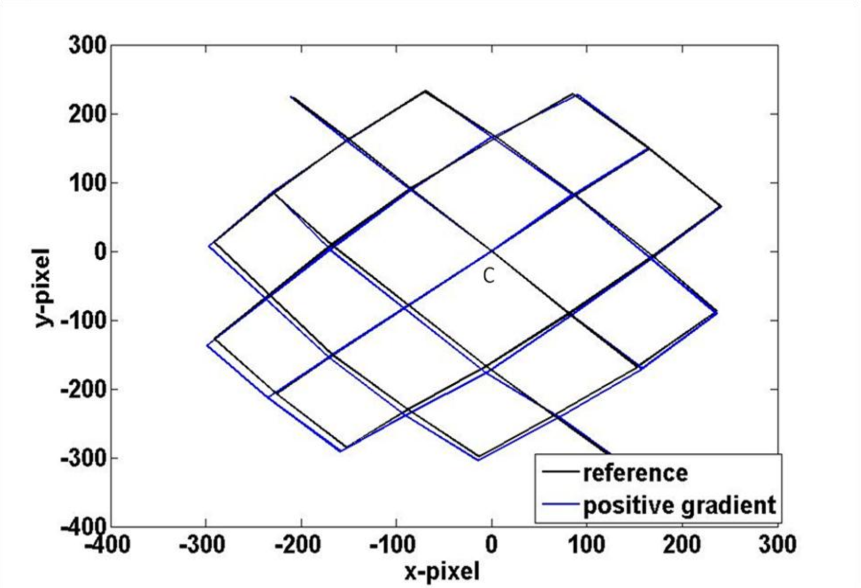


Shifts in the grid first appear near the boundary as already noted.

The grid shifts in the direction of lower temperature, i.e., larger density.

Grid Shift Upon Removal of Uniform Shift



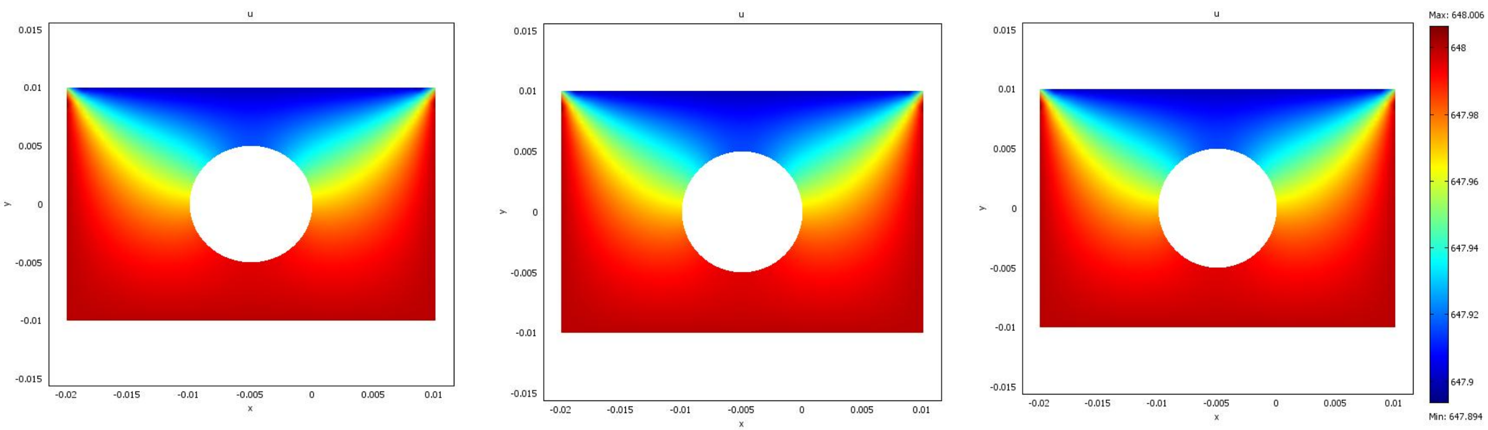


The shift in the center region is uniform for both positive and negative temperature gradients. Hence the experiment provides a uniform density gradient in the bulk fluid (interior region).

Modeling

Numerical Results

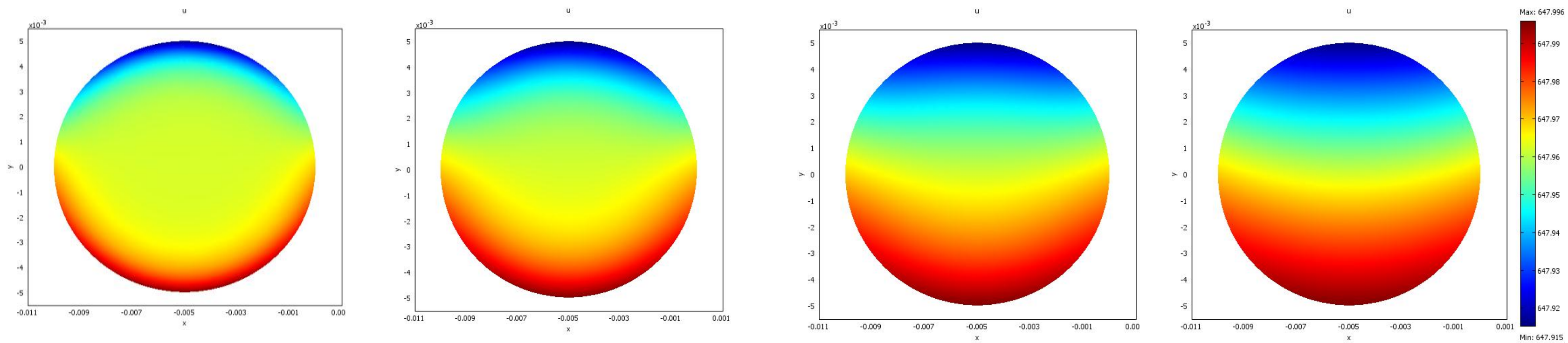
Configuration: 1 cm dia fluid cell in an inconel block
Initial Condition: T =648 K
Temperature Gradient: Decrease temperature of top surface by 0.1 K in 400 seconds



(a) 500 seconds **(b) 1000 seconds** **(c) 2500 seconds**

Cell block temperature equilibrates quickly

Numerical Results (contd.) : Temperature Field in the Fluid



(a) 400 seconds

Diffusive boundary layer + piston effect in bulk fluid

(b) 1000 seconds

Diffusive boundary layer extending into bulk; piston effect diminished

(c) 2500 seconds

Diffusive equilibration underway in entire cell

(d) 5000 seconds

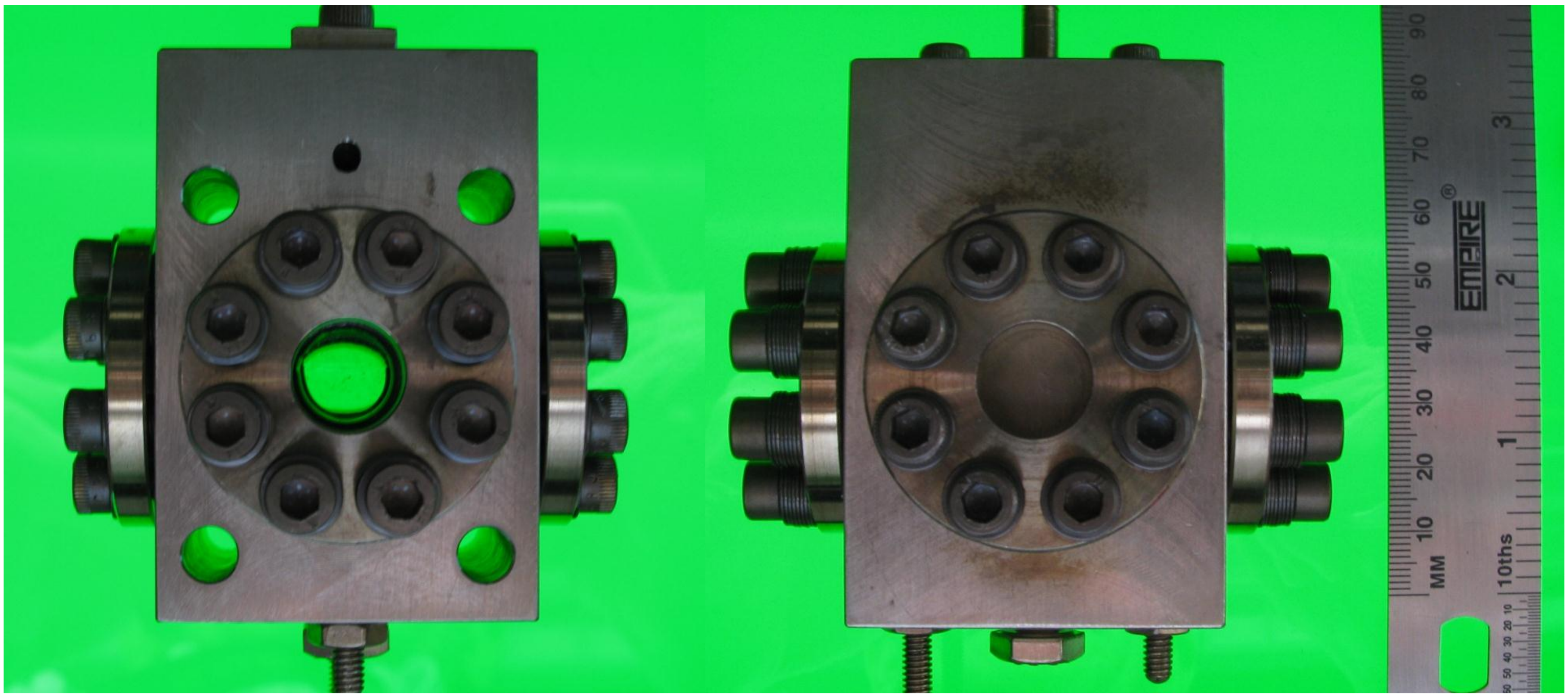
Diffusive equilibration complete

The temperature gradient in the bulk fluid is negligible at the early stages. When diffusive equilibration is complete there is a uniform temperature (and density) gradient in the central region (nearly linear temperature profile).

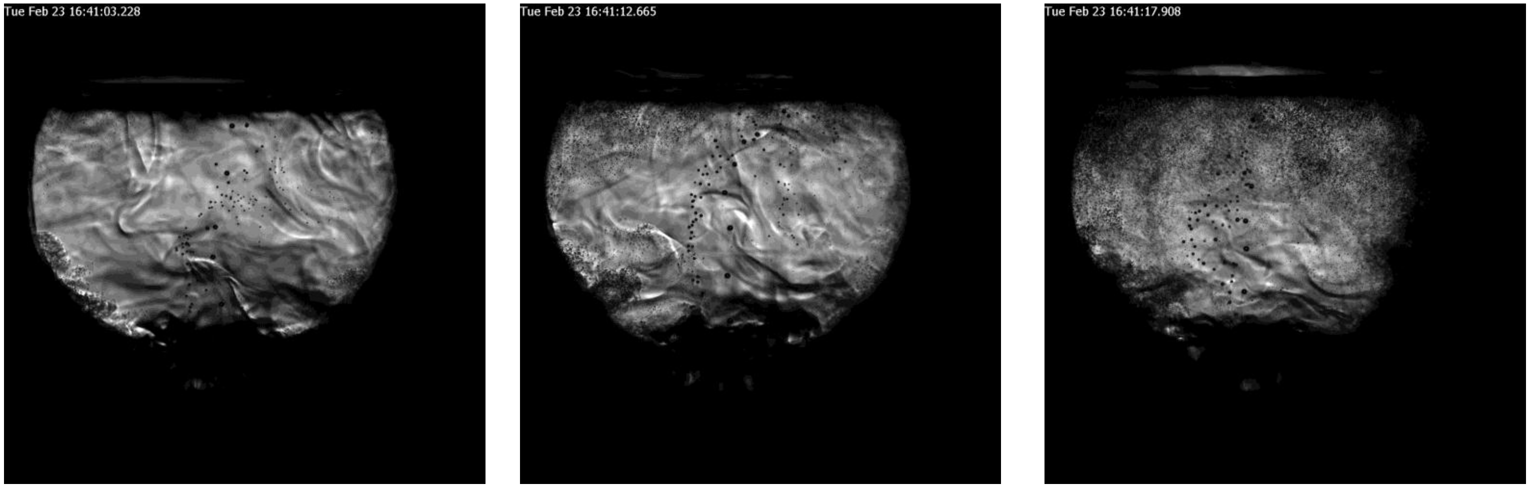
At equilibration, temperature gradient in the y-direction is greater than the gradient in the x-direction.

Ground Based Testing

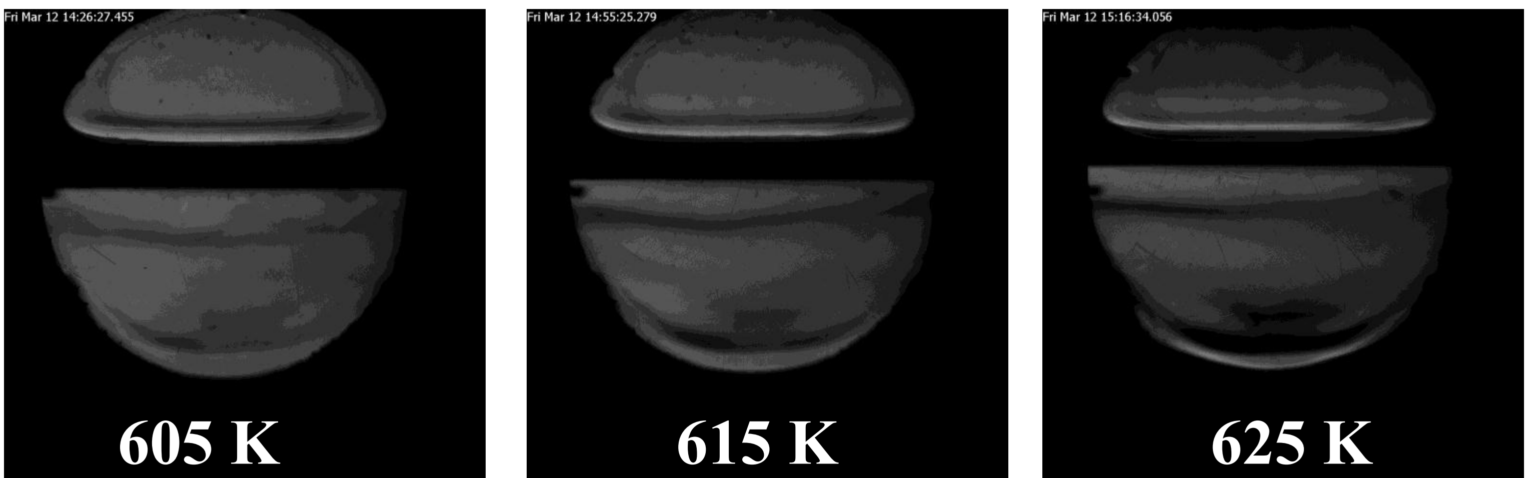
SCWM Lab Test Cell 1



Liquid water-vapor regions near the critical temperature

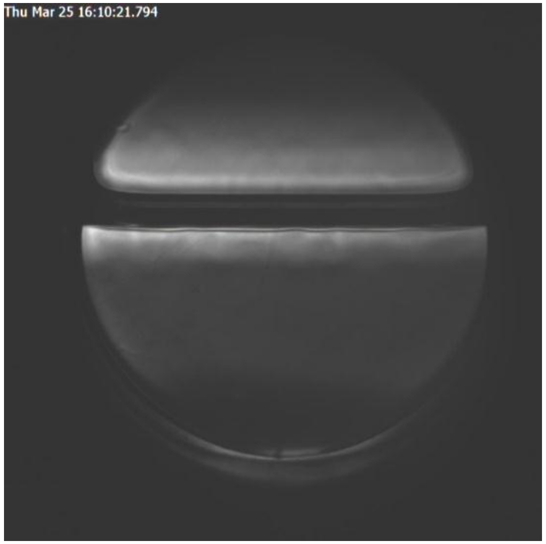


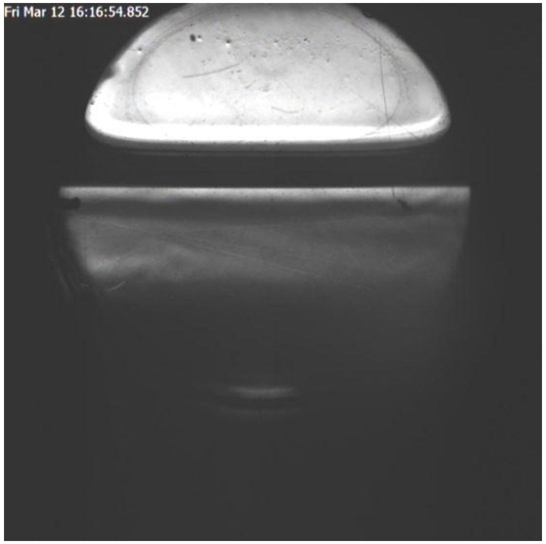
Liquid water-vapor regions during heat-up for a 10% Na2SO4 -water solution



Stratification during cool-down

Pure water | **10%-w sodium sulfate**





Summary - Preliminary Observations

- **The precipitated salt particulates/agglomerates are on the order of a 10-20 microns.**
- **Stratification/banding, suggesting a salinity concentration "inversion", occurs during quenching**

ACKNOWLEDGMENTS

- **NASA HQ (Exploration Science Mission Directorate)- F. Chiaramonte, Program Executive**

- **NASA GRC (Space Flight Systems Directorate)-**

  F. Kohl, ISS Research Project Manager

- **National Center for Space Exploration Research-**

  D. Gotti, J. Owens, W. Yanis